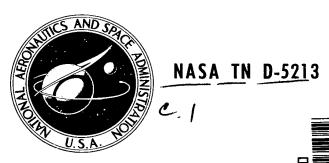
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RADIATION-INDUCED REDUCTION OF AMMONIACAL NICKEL(II) SULFATE TO METALLIC NICKEL

by Warren H. Philipp and Stanley J. Marsik Lewis Research Center Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION . WASHINGTON, D. C. . MAY 1969



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ABSTRACT

Aqueous ammoniacal nickel(II) sulfate solutions containing a variety of soluble organic compounds were irradiated with 2-MeV electrons. When this organic additive was either a primary alcohol, secondary alcohol, or cyclic ether, the divalent nickel was reduced to elemental nickel powder. The reaction mechanism involves the decomposition of water by the ionizing radiation into hydrogen atoms and hydroxyl radicals. The organic additive scavenges the hydroxyl radicals by reducing them, and the remaining hydrogen atoms then reduce the divalent nickel to the free metal powder.

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SUMMARY

Aqueous ammoniacal nickel(II) sulfate solutions containing a variety of soluble organic compounds were irradiated with 2-million volt electrons. When this organic additive was either a primary alcohol, secondary alcohol, or cyclic ether, the divalent nickel was reduced to elemental nickel powder. The highest nickel yield, G(Ni) of 0.88 atom per 100 eV energy, was obtained with the secondary alcohol, 2-propanol. Other organic additives, tertiary alcohol, ketone, acetylenic alcohol, aldehyde, and polyhydroxyl alcohol, caused little or no nickel to form.

The reaction mechanism involves the decomposition of water by the ionizing radiation into hydrogen atoms and hydroxyl radicals. The organic additive scavenges the hydroxyl radicals by reducing them, and the remaining hydrogen atoms then reduce the divalent nickel to the free metal powder.

Certain organic compounds such a formaldehyde inhibit nickel formation. The yield of nickel is also hindered by high ammonia concentration. It is concluded that organic nitrogen compounds formed by the reaction of the organic compound with ammonia scavenge the hydrogen atoms necessary for the reduction of divalent nickel. Because of this, the use of many good organic hydroxyl radical scavengers results in practically no nickel in ammoniacal systems. The reactions of organic nitrogen compounds with hydrogen are probably catalyzed by small amounts of newly formed nickel powder.

Unusually high nickel yields, G(Ni) of 11.5, are obtained in ammoniacal solutions containing the strongly reducing hypophosphite ion. In this case, the electron beam is believed simply to supply the activation energy for the reaction to occur.



INTRODUCTION

This report is an extension of our work on the formation of copper metal (Cu) by radiation chemistry (using 2-MeV electrons from a linear accelerator). The present work is concerned with the preparation of the more active metal nickel (Ni) by the same method. Because of the similarity of general conditions (e.g., the use of solutions where water is the main constituent), the general reaction mechanism postulated for Cu formation is assumed to apply also to the preparation of Ni.

The radical-scavenging mechanism described in the previous report (ref. 1) can be summarized as follows: The initial step is the decomposition of water (H_2O) by the ionizing radiation into hydrogen (H) atoms and hydroxyl (OH) radicals:

$$H_2O + Energy \rightarrow H \cdot + OH \cdot$$

Next, an organic additive scavenges the OH radicals by reducing them:

Additive + OH·
$$\rightarrow$$
 H₂O + Oxidized additive

The remaining H atoms then reduce the metal ion to the free metal.

Chemical reactions pertaining to Ni formation are considerably more complicated than those for Cu deposition. The greater complexity of reactions involved in Ni formation stems from two intrinsic properties of Ni: (1) Ni is an active metal, above hydrogen in the electromotive series of elements, and (2) Ni is a good catalyst for reactions involving hydrogen.

Because Ni is an active metal, it tends to reduce H ions. To suppress this reduction, the use of solutions of high pH is desirable. This can be accomplished conveniently by using an ammoniacal system. Thus, this report is chiefly concerned with the radiation-induced reduction of the hexamminenickel(II) ion $Ni(NH_3)_6^{+2}$ to elemental Ni.

The main purpose of the report is to demonstrate that Ni can be produced in good yield through radiation chemistry of aqueous solutions, as well as to show the effect of various parameters on Ni yield. The report discusses (1) the effectiveness of various organic compounds as hydroxyl radical scavengers for Ni formation, (2) the inhibition effect of certain organic compounds on the Ni yield in the aqueous nickel(II) sulfate (NiSO₄) - ammonia (NH₃) - 2-propanol system, (3) the results of varying concentration parameters on the Ni yield in the aqueous NiSO₄ - NH₃ - methanol system, and (4) the results of experiments using the strongly reducing inorganic additive sodium hypophosphite.



EXPERIMENTAL PROCEDURE

The nickel sulfate hexahydrate and ammonium hydroxide used were ACS grade. The amminenickel(II) complex ion $\mathrm{Ni(NH_3)}_6^{+2}$ forms on addition of ammonium hydroxide (NH₄OH; aqueous ammonia, NH₃) to the nickel salt solution.

The organic additives (scavengers and inhibitors) were selected to cover a variety of structures and functional groups. The selection was limited to compounds that are soluble in the ammoniacal nickel(II) sulfate solution and that do not by themselves reduce divalent nickel. These organic additives included formaldehyde (CH₂O), methanol (CH₃OH), ethanol (C₂H₅OH), 2-propanol ((CH₃)₂CHOH), 2-propyn-1-ol (C₃H₃OH), glycerine (C₃H₅(OH)₃), acetone ((CH₃)₂CO), 1-butanol (C₄H₉OH), 2-methyl-2-propanol ((CH₃)₃COH), tetrahydrofuran (C₄H₈O), and mannitol (C₆H₈(OH)₆). Also, the inorganic additive sodium hypophosphite (NaH₂PO₂) was used. All chemicals were high purity and were used without further purification. Prior to irradiation, the specific amount of the additive was dissolved in the nickel salt solution.

The reaction vessel (fig. 1) and general procedure are described in the earlier report on copper (ref. 1). As in the experiments with copper, the nickel solution was agitated during irradiation by passing helium through a glass frit at the base of the reaction vessel. The contents of the reaction vessel were kept at about 20° C during irradiation by immersing the vessel in a water bath. Because NaH₂PO₂ will reduce the hot amminenickel(II) complex, solutions containing this additive were kept at 5° C during irradiation. An arbitrary choice of 2-million volt electrons for all irradiations proved to be convenient for ease in using the equipment. Because of practical considerations, such as heat generation and irradiation time, a beam current of 15 to 20 microamperes was selected. The total dose was either 0.12 or 0.18 coulomb.

After irradiation was completed, the insoluble nickel powder was removed by centrifuging and washed with dilute aqueous ammonia. The nickel powder was suspended in acetone, filtered on a fritted glass filter, and dried at room temperature overnight. The filter containing the nickel was then further dried at 80° C for about 2 hours and weighed. A relatively low drying temperature was used to prevent oxidation of the metal powder. The metal powders were ferromagnetic. The product was identified by X-ray diffraction and by chemical analysis (measuring the weight increase caused by igniting the Ni to nickel oxide (NiO)).

All values of nickel yield are expressed in the commonly used term G(Ni), atoms of Ni formed per 100 eV energy input. The term G(Ni) is calculated from the quantity of Ni formed, the energy of the electron (2 MeV), and the accumulated dose read on the beamcurrent integrator. All runs were made in duplicate, and the results are the average of two runs. Precision of the results was about 10 percent.

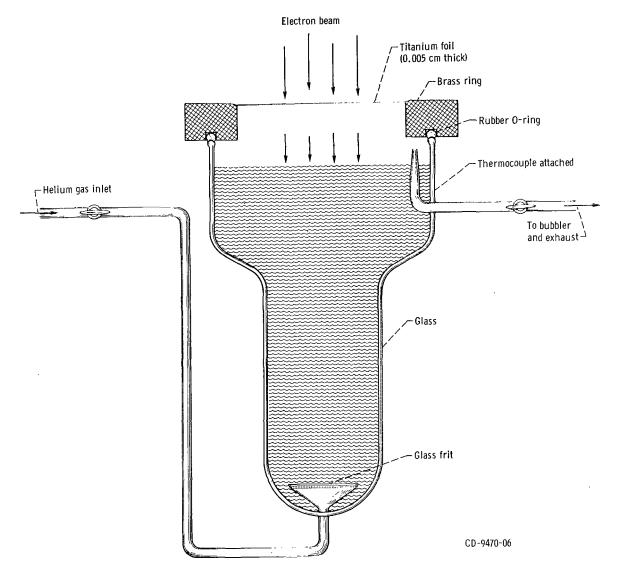


Figure 1. - Irradiation vessel.

RESULTS AND DISCUSSION

Experiments Using Organic Additives

Because of the tendency of Ni to reduce H^+ (ref. 2, p. 341), the best nickel yields are expected in solutions of high pH. This advantage of high pH for Ni formation was demonstrated experimentally. Irradiation of neutral aqueous 0.2 molar $NiSO_4$ (measured pH of about 7) containing either 1 molar methanol or 1 molar 2-propanol gave a nickel yield G(Ni) of only 0.077 and 0.066, respectively. The presence of 2 molar NH_3 in the

TABLE I. - EFFECT OF ORGANIC ADDITIVES ON YIELD OF FREE NICKEL FROM

AQUEOUS NICKEL(II) SULFATE - AMMONIA SOLUTIONS

[Electron energy, 2 MeV; beam current, 20 µA.]

Type of additive	Additive	Additive concentration, moles/liter	'Nickel sulfate concentration, moles/liter	Ammonia concentration, moles/liter	Dose,	Nickel yield, G(Ni), atoms/100 eV
Secondary alcohol	2-Propanol, C ₃ H ₇ OH	1,0	0.05	1.0	0.12	0.83
		1.0	. 2	2.0	. 18	. 88
		1.0	. 2	2.0	. 12	. 17
Primary alcohol	Methanol, CH ₃ OH	1.0	0.05	1.0	0.12	0. 20
		2.0	. 2	2.0	. 12	. 18
		2.0	. 2	2.0	. 18	. 23
	Ethanol, C ₂ H ₅ OH	1.0	0. 2	2.0	0. 18	0. 22
	1-Butanol, C ₄ H ₉ OH	1.0	0.2	2.0	0.18	0. 24
Cyclic ether	Tetrahydrofuran,	1.0	0.05	1.0	0.12	0. 16
	C ₄ H ₈ O	1.0	. 2	2.0	. 18	. 16
Tertiary alcohol	2-Methyl-2-propanol,	1.0	0.05	1.0	0.12	0.04
	(СН ₃) СОН	1.0	. 2	2.0	. 18	. 03
Polyhydroxyl alcohol	Glycerine, C ₃ H ₅ (OH) ₃	1.0	0.05	1.0	0.12	<0.01
		1. 0	. 2	2. 0	. 18	<. 01
	Mannitol, C ₆ H ₈ (OH) ₆	0. 5	0. 2	2. 0	0. 18	0.04
Acetylenic alcohol	2-Propyn-1-ol, C ₃ H ₃ OH	1.0	0. 2	2. 0	0. 18	<0.01
Aldehyde	Formaldehyde, CH ₂ O	1.0	0. 2	4.0	0. 18	<0.01
Ketone	Acetone, CH ₃ COCH ₃	1.0	0. 2	2. 0	0. 18	<0.01

same solution (measured pH of about 12) resulted in increased G(Ni) values of 0.17 and 0.88 for the same additives. In the interest of obtaining the best possible Ni yield, no further study of neutral $NiSO_4$ was made.

Table I presents the nickel yield from ammoniacal NiSO₄ containing a variety of organic compounds. The highest nickel yields, G(Ni) of about 0.85, were obtained using the secondary alcohol, 2-propanol. Somewhat lower values, G(Ni) of about 0.20, resulted from the use of primary alcohols and the cyclic ether, tetrahydrofuran. The rest of the organic additives, tertiary alcohol, polyhydroxyl alcohol, acetylenic alcohol, aldehyde, and ketone gave little or no nickel. Because tertiary alcohols and ketones are not reducing, they cannot scavenge hydroxyl radicals, and no Ni formation is expected with these additives. The fact that practically no Ni was produced with the acetylenic alcohol

is attributed to the hydrogen atom scavenging ability of the acetylenic bond. However, the other nil yields of Ni were unexpected because they differed from the results of copper deposition from aqueous copper(II) sulfate (CuSO₄) (no NH₃ added). Values of copper yield G(Cu) of about 2.2 were obtained for essentially all reducing organic additives (those containing one or more hydrogen atoms on the carbon atom comprising the functional group, ref. 3).

Figure 2 shows the effect of NH_3 concentration on Ni yield for a solution containing 0. 2 molar $NiSO_4$ and 1 molar CH_3OH . The nickel yield G(Ni) is at its highest value of

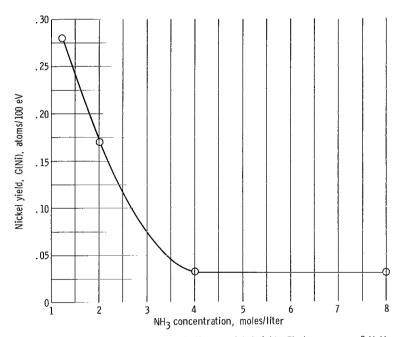


Figure 2. – Effect of $\rm NH_3$ concentration on nickel yield. Electron energy, 2 MeV; beam current, 20 microamperes; dose, 0.12 coulomb; $\rm NiSO_4$ concentration, 0.2 molar; $\rm CH_3OH$ concentration, 1.0 molar.

0. 28 at the lowest NH_3 concentration (about 1.2 molar) necessary to prevent precipitation of divalent Ni . This amount of NH_3 is required for the complete conversion of $\mathrm{Ni}(\mathrm{H}_2\mathrm{O})_6^{+2}$ (the aquonickel(II) ion) to $\mathrm{Ni}(\mathrm{NH}_3)_6^{+2}$. The presence of excess NH_3 above this amount decreases $\mathrm{G}(\mathrm{Ni})$ to a value of 0.031 at about 4 molar NH_3 . It is apparent that this excess NH_3 inhibits Ni formation at least when methanol is the organic additive.

A further understanding of the reason for NH_3 inhibition and an explanation as to causes for nil yields of Ni with certain good organic reducing agents, aldehydes, and polyhydroxyl alcohols were provided by experiments to test inhibition effects of certain organic compounds on $\mathrm{G}(\mathrm{Ni})$ in ammoniacal NiSO_4 containing 2-propanol. The secondary

TABLE II. - EFFECT OF SECOND ORGANIC ADDITIVE
ON YIELD OF NICKEL FROM AQUEOUS NICKEL(II)
SULFATE - AMMONIA - 2-PROPANOL SOLUTIONS

[Electron energy, 2 MeV; beam current, 20 μ A; dose, 0.12 C; NiSO₄ concentration, 0.1 M; NH₄OH concentration, 2.0 M; 2-propanol concentration, 0.5 M.]

Additive	Additive concentration moles/liter	Nickel yield, G(Ni), atoms/100 eV	
None		0.82	
Glycerine	1.0	0.014	
Formaldehyde	0.5	0. 18	
Methanol	1.0	0.52	
Acetone	0.5	0.46	
	1.0	. 43	

alcohol, 2-propanol, system was selected as the basis of these runs because this additive gave the highest Ni yield of all the organic additives used. The results of these experiments concerning the effect of the second additive on G(Ni) are given in table II. All the second additives tested, methanol, acetone, and to a greater extent, formaldehyde and glycerine, decrease G(Ni) from its highest value of 0.82. It should be noted here that acetone is the oxidation product of 2-propanol, and formaldehyde is one of the oxidation products of methanol. We believe that the greater inhibition of formaldehyde on nickel formation compared with that of acetone is the cause for the lower nickel yield obtained with methanol (G(Ni) of about 0.2) as compared with that obtained for 2-propanol (G(Ni) of about 0.8) (see table I).

Acetone does not readily form addition products with NH $_3$ (ref. 4, p. 78). On the other hand, formaldehyde, the oxidation product of methanol, reacts readily with aqueous NH $_3$ to form the condensation product hexamethylenetetramine ((CH $_2$) $_6$ N $_4$) (ref. 4, p. 67). Apparently, further oxidation of formaldehyde in the presence of NH $_3$ produces some formamide. The characteristic odor of formamide was noted in experiments with methanol and formaldehyde. Based on these considerations and the results in figure 2 showing the inhibition effect of NH $_3$, these nitrogen-containing organic compounds were assumed to reduce the yield of Ni by actually scavenging some H atoms. Such H atom scavenging reactions may be catalyzed by the previously formed Ni powder. As shown in table II, methanol itself does not appreciably inhibit the formation of Ni because, even in

its weak addition product with NH₃, it is not an oxidizing agent and thus cannot scavenge H atoms.

Surprisingly, glycerine, a good OH radical scavenger for Cu deposition, strongly impedes Ni formation in the presence of 2-propanol. In this case, apparently, a reaction takes place which involves consumption of H atoms. Such a hydrogenation reaction may be catalyzed by small amounts of Ni that form in the reaction zone. The mechanism for mannitol (table I) is likely similar to that for glycerine.

Because of the complexity of reactions for the ${\rm NiSO_4}$ - ${\rm NH_3}$ - ${\rm CH_3OH}$ - ${\rm H_2O}$ system as compared with that of the ${\rm CuSO_4}$ - ${\rm CH_3OH}$ - ${\rm H_2O}$ system, the dependencies of G(Ni) on ${\rm NiSO_4}$ and ${\rm CH_3OH}$ concentrations are more difficult to evaluate than those for G(Cu). For this reason, a satisfactory explanation of all trends in the curve of G(Ni) against ${\rm CH_3OH}$ concentration (fig. 3) and the curve of G(Ni) against ${\rm NiSO_4}$ concentration (fig. 4)

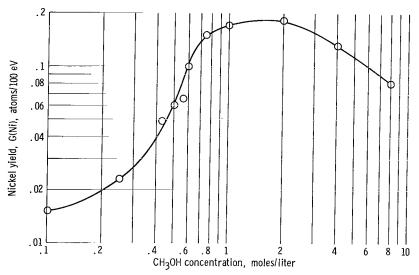


Figure 3. - Effect of $\mathrm{CH_3OH}$ concentration on nickel yield. Electron energy, 2 MeV; beam current, 20 microamperes; dose, 0.12 coulomb; $\mathrm{NiSO_4}$ concentration, 0.2 molar; $\mathrm{NH_3}$ concentration, 2 molar.

cannot be given at this time. Figure 3 shows G(Ni) over a wide range of CH_3OH concentration (0. 1 to 8.0 M) for a solution containing 0.2 molar $NiSO_4$ and 2 molar NH_3 . The maximum value of G(Ni) of about 0.18 is reached near 2 molar CH_3OH and then drops to 0.08 at 8 molar CH_3OH . Decreasing the CH_3OH concentration from 1 to 0.1 molar decreases G(Ni) rapidly from 0.17 to 0.015. This dependence suggests a kinetic relation of the nickel yield on CH_3OH concentration. Such a dependence in this region may involve the scavenging of OH radicals by CH_3OH as a rate-controlling reaction step:

$$CH_3OH + 2OH \cdot \rightarrow HCOH + 2H_2O$$

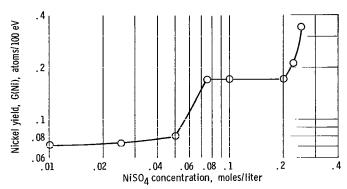


Figure 4. - Effect of NiSO₄ concentration on nickel yield. Electron energy, 2 MeV; beam current, 20 microamperes; dose, 0.12 coulomb; CH₃OH concentration, 1.0 molar; NH₃ concentration, 2.0 molar

In figure 4, G(Ni) is plotted as a function of NiSO $_4$ concentration (0.01 to 0.20 M) in a solution containing 1 molar CH $_3$ OH and 2 molar NH $_3$. In a limited NiSO $_4$ concentration range (0.075 to 0.20 M), G(Ni) is constant and in this range may be compared with a similar plot for the formation of copper from aqueous CuSO $_4$. However, G(Cu) remained practically constant over a much wider CuSO $_4$ concentration range (0.05 to 1.25 M CuSO $_4$). The lack of dependence of G(Cu) on CuSO $_4$ concentration implied that the reaction of the divalent copper ion with the reducing species, H atoms, was not the rate-controlling step in the overall mechanism. Similar reasoning may apply to Ni formation in the NiSO $_4$ concentration range 0.075 to 0.20 molar; the reduction of divalent nickel by H atoms is not rate controlling.

Above 0. 20 molar NiSO_4 , $\operatorname{G(Ni)}$ increase abruptly from 0. 17 to a maximum value of 0. 34 at 0. 25 molar NiSO_4 . No higher NiSO_4 concentration was used because of the precipitation of divalent nickel above this concentration. This abrupt increase in $\operatorname{G(Ni)}$ is ascribed for the most part to a decrease in NH_3 concentration as a result of its complexing with the added concentration of divalent nickel ion according to the equation

$$Ni(H_2O)_6^{+2} + 6NH_3 - Ni(NH_3)_6^{+2} + 6H_2O$$

Thus, the uncomplexed $\mathrm{NH_3}$ concentration remaining after the addition of 0.25 molar $\mathrm{NiSO_4}$ to 2.0 molar $\mathrm{NH_3}$ is 0.5 molar. At the foot of the rise at 0.2 molar $\mathrm{NiSO_4}$, the uncomplexed $\mathrm{NH_3}$ concentration is 0.8 molar. In figure 2, this excess $\mathrm{NH_3}$ concentration range corresponds to the portion of the curve from 1.7 to 2 molar; the nickel yield is quite sensitive to $\mathrm{NH_3}$ concentration in this range.

Below 0.075 molar $NiSO_4$, the Ni yield decreases rapidly and then levels off beyond 0.05 molar $NiSO_4$. No explanation of this portion of the curve is attempted at this time.

Experiments Using Sodium Hypophosphite

In an effort to find the most effective OH radical scavenger affording the highest possible nickel yield from aqueous systems, sodium hypophosphite (NaH $_2$ PO $_2$) was selected because of the strong reducing potential of H $_2$ PO $_2^-$ in basic solution:

It is not surprising to find that the divalent nickel species is readily reduced to elemental Ni when an aqueous solution containing 0.2 molar NiSO₄, 2 molar NH₃, and 0.5 molar NaH₂PO₂ is boiled. At room temperature, however, no Ni forms after 48 hours. Irradiation of such a solution below 5° C by using a low beam current (5 μ A) and a small total dose (0.03 C) to prevent local overheating of the solution resulted in an unusually high nickel yield, a G(Ni) of 11.5. Unfortunately, the Ni obtained in this experiment contained a small amount of black powder insoluble in hot dilute hydrochloric acid. This insoluble material was probably a phosphide of nickel.

Because of the high value of G(Ni) obtained with NaH_2PO_2 and the strong reducing properties of this compound, we believe that the electron beam simply supplies the activation energy necessary for the reaction to occur and does not necessarily involve the radical scavenging mechanism proposed for the reducing organic additives.

CONCLUDING REMARKS

Because nickel powder can be produced in good yield by radiation chemistry of aqueous alkaline (ammoniacal) solutions, it is reasonable to suppose that other active metals (e.g., cadmium and iron) can likewise be produced. In ammoniacal nickel(II) sulfate solution, the highest nickel yields were obtained with a secondary alcohol, 2-propanol, as the organic additive. Other secondary alcohols are assumed to be as effective, not only for nickel formation but also for the preparation of other metals from ammoniacal solutions. On oxidation, secondary alcohols form ketones that are resistant to further oxidation and do not readily form addition products with ammonia (ref. 4, p. 78) as do aldehydes, the oxidation products of primary alcohols (ref. 4, p. 71). These organic nitrogen-containing addition products are believed to scavenge the hydrogen atoms necessary for the reduction of the metal ion to the free metal. Thus, it is reasonable to expect that in ammoniacal solutions primary alcohols would give poorer

yields of metals than secondary alcohols. This may not be the case, however, in non-ammoniacal basic solutions such as those containing sodium hydroxide.

Another consideration to bear in mind is that nickel is a good catalyst for reactions of hydrogen with organic compounds. Such reactions may well be enhanced by the presence of newly formed nickel powder in the reaction zone. It is therefore conceivable that primary alcohols would give relatively better yields of metals that are less catalytically active.

In addition to reactions involving the radical-scavenging mechanism, radiation may be used to promote thermodynamically feasible chemical reactions by supplying the necessary activation energy. Such a process may be illustrated by the formation of nickel in hypophosphite solutions. For such reactions, high-energy yields are expected because reactions of this type are theoretically spontaneous and tend to be self-propagating.

Lewis Research Center,

National Aeronautics and Space Administration, Cleveland, Ohio, March 18, 1969, 129-03-15-01-22.

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